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Prepared by	Xihui Sun/ Zhan Zhang			
Checked by	Zhenhui He			
Approved by	Zhenhui He			

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CHANGE TAKE BY HAND

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TABLE OF CONTENT

1. SCOPE 5

2. DOCUMENTS 5

2.1 5

3. AMS TTCS FILLING SYSTEM 6

3.1 Principle of the TTCS filling system 6
3.2 Structure of the portable TTCS filling system 6

4. MAIN TERM COMMENTARY OF THE TTCS FILLING PROCEDURE 8

5. PRINCIPLE OF VOLUME DETERMINATION 10

5.1 Volume determination by gas mass 10
5.2 Volume determination of the TTCS loop 10

6. TTCS FILL PROCEDURE 12

6.1 Preparation procedures 13
6.2 Leak rate of the TTCS loop 14
6.3 Determination TTCS volume and CO_2 mass to be filled 16
6.4 Fill a standard vessel with CO_2 from a Gas bottle 19
6.5 Calibration of the filled mass 20
6.6 Fill TTCS loop with CO_2 from SV1.5 22
6.7 Venting CO_2 from the TTCS loop 24
6.8 Temporary sealing the TTCS loop (FM) 26

7. FILLING PROCEDURE DATA SHEET 27

7.1 Preparation procedures 27
7.2 Filling system leak rate examination 28
7.3 Determination of filling system volume 30
7.4 Determination of TTCS volume 34
7.5 Fill a standard vessel with CO_2 from a Gas bottle 38
7.6 Calibration of the filled mass 43
7.7 Fill TTCS loop with CO_2 from SV1.5 47
7.8 Venting CO_2 from the TTCS loop 53

8. APPENDIX 55

8.1 Error analysis of volume determination of a vessel by weighting the gas mass inside 55

8.2 Error analysis of volume determination of TTCS loop by indirectly measuring the Nitrogen mass inside the loop 56

8.3 Error analysis of the total filled mass of TTCS loop 57

8.4 Calculation of the required filling mass into the SV1.5....................................59

8.5 Calculation example 60

LIST OF TABLES

Table 2-1 Reference... 5

Table 2 Commentary about main term of the TTCS filling procedure 8

Table 3 Preparation procedures... 13

Table 4 Procedures for leak rate examination... 14

Table 5 Procedure of determination of the volume of the TTCS loop V_{loop} and the CO_2 mass to be filled 16

Table 6 Procedure of determination of filling a standard vessel with CO2 from a gas bottle.. 19

Table 7 Procedure of Calibration of the filled mass... 20

Table 8 Procedure of filling TTCS loop with CO2 from SV1.5................................. 22

Table 9 Procedures for venting CO_2 from TTCS loop.. 25

Table 10 saturated properties of CO2 from 15°C to 29°C with interval of 1°C 62

LIST OF FIGURES

Figure 3-1 Portable filling system 6

Figure 3-2 Schematic TTCS filling system 7

Figure 5-1 Weight of gas mass of the filled standard vessel before filling TTCS, to determine the volume of TTCS LOOP 11

Figure 5-2 Weight of gas mass of the filled standard vessel after filling TTCS to determine the volume of TTCS FM 11

Figure 6-1 Schematic of venting CO_2 from TTCS loop 25
Figure 8-1 CO2 initial state and mass distribution between the filling system and the TTCS loop before starting filling 59
Figure 8-2 CO_2 finial state and mass distribution between the filling system and the TTCS loop after finishing filling 59
Figure 8-3 CO_2 mass distribution between the filling system and the TTCS loop after finishing filling 60

1. SCOPE

This document describes the AMS Tracker Thermal Control System (TTCS) filling system, and the CO_2 filling procedure for TTCS FM after integration, based on this filling system.

2. DOCUMENTS

2.1

Table 2-1 Reference

RD-1	TTCS Filling System and Accuracy, by G. van Donk/J. van Es	NLR-Memorandum AMSTR-NLR-TN-019-Issue 02	
RD-2	Cleaning of 316L tubes and components, by WJ Xiao	TTCS-SYSU-GS-TN-001-1.0	
RD-3	Leak detection for TTCS EM by He mass spectrography, JF Ding	TTCS-SYSU-GS-RP-003-1.0	

3. AMS TTCS FILLING SYSTEM

3.1 Principle of the TTCS filling system

The TTCS filling system is designed to fill the required CO_2 into the TTCS loop with mass accuracy of 4%. The filling system is capable to measure the TTCS loop volume, which allows the determination of the fill mass of CO_2.

The requirement details and the filling principle can be referred to RD1 “TTCS Filling System and Accuracy”.

3.2 Structure of the portable TTCS filling system

Figure 3-1 Portable filling system

Figure 3-1 and figure 3-2 shows the structure and the principle of the TTCS filling system. The filling system consists of three standard vessels with about volume of 1.5 liters (labeled as: VS_A, VS_B, and VS_C, respectively), a filling box (see Figure 3-1, the photo above, and Figure 3-2), a vacuum pump, and an electronic balance (locally supplied for the last two), a digital pressure gauge, and flexible extension filling tubes. The filling box consists of a vacuum valve and valve 4 that connect to the vacuum connector, a vacuum gauge to measure the vacuum, valve 1 that connects to the TTCS loop through connector 1, valve 2 that connects to the gas bottle through connector 2, valve 3 that connects to a standard vessel through connector 3, a digital pressure gauge to measure filling pressure, a finer gauge mount on the front panel measures outlet pressure of pressure regulator and these two pressure gauges can measure pressure of TTCS indirectly during filling. And two filters that prevent micro-particles from entering the filling system (including standard vessels) and the TTCS loop.

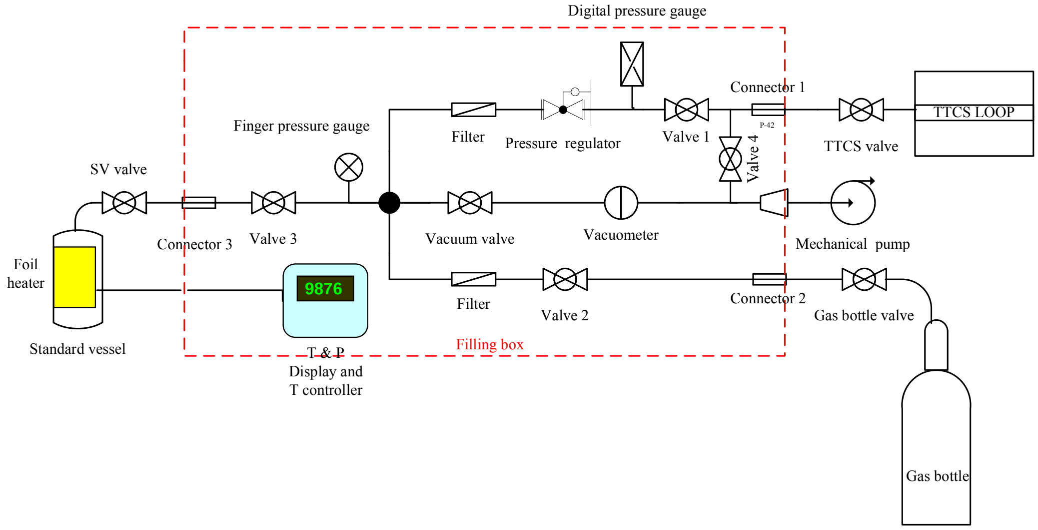

Figure 3-2 Schematic TTCS filling system

4. MAIN TERM COMMENTARY OF THE TTCS FILLING PROCEDURE

Table 2 Commentary about main term of the TTCS filling procedure

Nomenclature	
T	Temperature
M	Mass
V	Volume
ρ_G	Density of gas
Subscript	
C	Calculated
E	Empty
F	Filled
FT	Filling tube and box
FR	Fill rate (g/L)
Filled_VS	The filled standard vessel
Filled_L	The filled loop
L	The TTCS Loop
O	Obtained
R	Retained
VS	Standard vessel with volume of 1.5L

T_{room}	Clean room temperature
M_{C_L}	**$=V_{Loop} \times FR$** Calculated CO_2 mass to be filled into the TTCS LOOP
ρ_G (T, P)	CO_2 density inside the standard vessel determined by the chosen final temperature of the standard vessel and the final saturated pressure of the TTCS loop during filling
$M_{C\_R\_VS}$	**$=V_{VS} \times \rho_G$ (T)** The calculated CO_2 mass to be retained in the SV1.5 after filling
$M_{C\_R\_FT}$	**$=V_{FT} \times \rho_G$ (T)** The calculated CO_2 mass to be retained in the filling box and the extend tube after filling
M_{C_R}	**$=M_{C\_R\_VS} + M_{C\_R\_FT}$**
M_{E_VS}	Net mass of the SV1.5 in 15Pa.
M_{C_VS}	**$= M_{E\_VS} + M_{C\_L} + M_{C\_R}$;** Total calculated mass of the filled SV1.5 before filling to the TTCS Loop
M_{Filled_VS}	The obtained gross mass of CO_2 in the SV1.5 before filling
M_{O_VS}	**$= M_{Filled\_VS} - M_{E\_VS}$** The obtained net mass of CO_2 to be filled into the SV1.5 before filling
$M_{O\_R\_VS}$	The obtained CO_2 mass retained in the SV1.5 after filling
$M_{O\_R\_FT}$	The obtained CO_2 mass retained in the filling tube after filling
M_{Filled_L}	$= M_{O\_VS} - M_{O\_R\_VS} - M_{O\_R\_FT}$ The obtained CO_2 mass filled into the TTCS Loop

5. PRINCIPLE OF VOLUME DETERMINATION

5.1 Volume determination by gas mass

Assumption: There is no leakage of the measuring system, including the containers (here components and couplings), during the period of measurement; and the temperature can be controlled with uniformity within 2.0K; no nitrogen absorption on the inner surface of the system (here TTCS loop) that made of stainless steel.

A container, with mass of M_E and filled N_2 of M_{N2}, is connected to another container with unknown volume V_2, after measuring the N2 mass filled into V2 by weighting, and the final pressure by employing the filling system with high precision pressure sensor, and the temperatures of the containers, the unknown volume of V_2 can be determined by using the ideal gas equation.

5.2 Volume determination of the TTCS loop

Fill nitrogen with the volumes of the known container to a pressure of 4.5MPa; weight the mass of the container $M_{nitrogen1}$. Connect the container to a vacuumed loop and release nitrogen to the loop. Weight the mass of the container again $M_{nitrogen2}$, the nitrogen mass filled into the loop can then be calculated: $M_{nitrogen} = M_{nitrogen1} - M_{nitrogen2}$,

$$V_{Loop} = \frac{M_{nitrogen} R T_2}{P_2 m_{nitrogen}}$$

Here $m_{nitrogen}$ is molecular weight of nitrogen.

$\Delta M_B = \pm 0.1$g, and $\Delta T_{loop} = \pm 2$K, the error $\frac{\Delta V_{loop}}{V_{loop}} = \frac{\Delta P_{loop}}{P_{loop}} + 2\frac{\Delta M_B}{M_{He}} + \frac{\Delta T_{loop}}{T_{loop}}$ is about 2.4%.

Please refers to 8.2 for details.

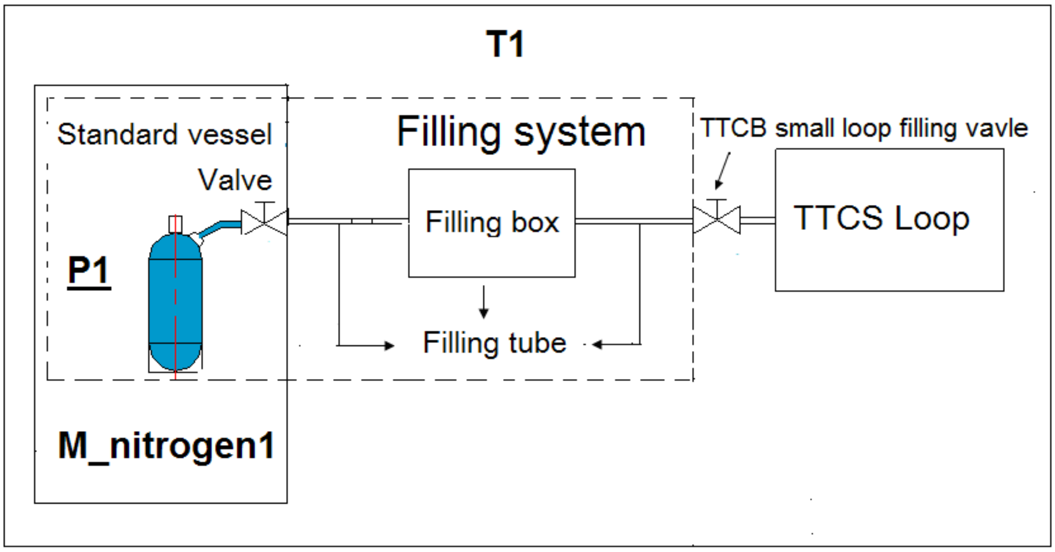

Figure 5-1 Weight of gas mass of the filled standard vessel before filling TTCS, to determine the volume of TTCS LOOP

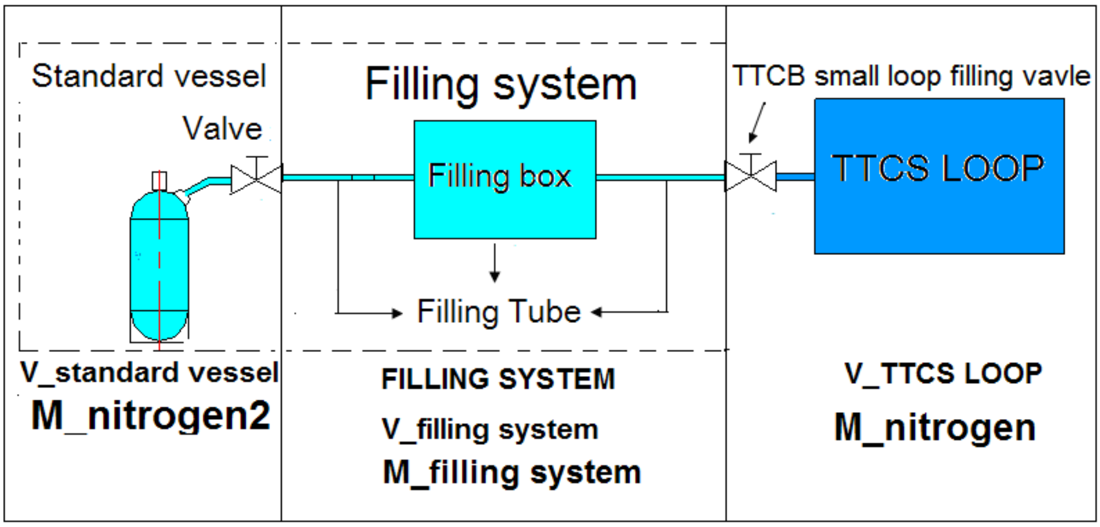

Figure 5-2 Weight of gas mass of the filled standard vessel after filling TTCS to determine the volume of TTCS FM

6. TTCS FILL PROCEDURE

The fill procedure consists of the following major steps:

1. Determination of the exact TTCS-loop volume, including that of the accumulator

2. Filling of the TTCS-loop with the exact CO_2 mass

Main implication of the filled TTCS is that TTCS-loop below 60°C (the maximum design temperature) is below 160bar, the maximum design pressure. We use a CO_2 filling system to fill the TTCS loop (including all the components), which can reach the required filling accuracy.

In general, the filling procedures include:

1. Preparation;
2. Leak test of the filling system, including extend tubes, and the TTCS loop;
3. Filling a standard vessel and the calibration of the filled mass before loop filling
4. Determination of the volume of the TTCS loop and the CO_2 mass to be filled.
5. Filling a standard vessel and then Filling a TTCS loop
6. Verify the fill mass of CO2 into the loop (by weighting the mass of the standard vessel)
7. Temporary Sealing of the TTCS loop
8. If refilling is required, CO2 venting from the TTCS.

6.1 Preparation procedures

Table 3 Preparation procedures

Step	Action
1.	
2.	Calibrate the volume of the filling box, the two connecting tubes and valves; (V_{FT}= 0.042 liter, if no additional extend tube is required)
3.	Select filling environmental temperature, e.g., 22°C;
4.	Wrap a heating tape around the filling tube outside the box;
5.	Prepare an electronic balance, with full scale of XXg and accuracy of 0.1g;
6.	Prepare a He-mass spectrometer to perform leak test
7.	Prepare a high precision APS (0.1%FS with FS=10MPa) for volume determination;
8.	Prepare pure N_2 gas bottle for volume calibration
9.	Prepare high purity (99.99%) CO_2 bottle for filling;
10.	Prepare a barrel with ice water mixture for cooling the vessel;
11.	Prepare a fan for cooling TTCS accumulator during filling;
12.	Connect a dry mechanical pump to the vacuum connector;

list of components to be prepared

6.2 Leak rate of the TTCS loop

In order to ensure the filling accuracy, the leak rate of the filling system need to be satisfied by the requirements below ***5×10^{-5} mbar·l/s***. Since the filling system had been used for several times, leak might be happened at the connectors of the filling system. Using equipment "He mass spectrometer" to check these connectors. If some connecting parts have a detectable leak, replace them with new ones.

To avoid the helium remaining in the filling system after the leak test, a flash operation is needed. N_2 could be taken to flash the filling system.

Table 4 Procedures for leak rate examination

Step	Action
1.	Seal the filling system with the extend tubes
2.	Measure the background leak rate of the "He mass spectrometer"
3.	Connect the He mass spectrometer to the vacuum connector
4.	Vacuum the filling system
5.	Measure the leak rate of the filling system without helium gas.
6.	Using ***negative*** method, blowing helium gas, to measure the leak rate of these connectors, connector 1, 2 and 3. Leak rate for each connector should be below ***5×10^{-5} mbar·l/s***.
7.	Disconnect the vacuum pump and measure the background leak rate of the equipment.
8.	**Flush the filling system**
9.	Connect the N2 bottle to the connector 2
10.	Pressurize the filling system to 40bar

11.	Release the N2 from the system
12.	Repeat above steps 10**Error! Reference source not found.** and **Error! Reference source not found.**11

If the leak rate of the filling system, which reaches ***5.0×10^{-5}mbar·l/s,*** are detected with a He-mass spectrometer, this gives a leak rate of 7.7×10^{-3}g/day/connector, which is neglectable as compared to 850g fill mass of CO_2.

NOTE: As the portable filling system is disconnected and reconnected before being used, the leak rate of the system must be detected again.

6.3 Determination TTCS volume and CO_2 mass to be filled

In Figure 4-1 and Figure 4-2 the set-up for the volume determination is shown. The principle of volume determination is to measure mass of a standard vessel before and after filling the TTCS-loop. The mass difference gives (in)-directly the TTCS loop-volume.

Note:

1. Because of assembly, pressure regulator does not function compatibly to control the filling speed, we decide to so by turning on and off valves, to replace operating the pressure regulator. Monitoring the DPS by TTCE at the same time (**not higher than 0.5 bar).**
2. Three temperature sensors of Pt-1000 of new filling system should be mounted at the top, the middle, and the bottom of the standard vessel's side face, which is enveloped by the heater as a jacket (No.1 PT1000 for control, No.2 PT1000 and No.3 PT1000 for monitor. During filling CO2 from the standard vessel to the TTCS loop, No.1 PT1000 should be paste on the middle of the standard vessel's side face, the rest PT1000 should be paste on the top and bottom of the standard vessel's side face).

Table 5 Procedure of determination of the volume of the TTCS loop V_{loop} and the CO_2 mass to be filled

Step	Action
1.	Take clean room temperature as T_{room}
2.	Connect the SV1.5 (anyone of stand vessel can be used) to connector 3; **<u>Note: Longer extend tube may be used, and height difference may exist between the SV1.5 and connector 3.</u>** **<u>The extend tube between connectors and valves must be mechanically supported to reduce strain.</u>**
3.	Connect a N_2 bottle to connector 2;

4.	Connect the TTCS Loop to connector 1; **Note: Longer extend tube may be used, and height difference may exist between the TTCS loop and connector 1.** **The extend tube between connectors and valves must be mechanically supported to reduce strain.**
5.	Close valve 1, valve 4 and the N_2 bottle valve, open valve 2, valve 3, SV1.5valve, pressure regulator the vacuum valve and the TTCS valve slowly, to pump SV1.5 down to 30Pa;
6.	Close the vacuum valve, open the valve of the N_2 bottle;
7.	Fill the SV1.5 with N_2 up to 4.5MPa, then close valve 2; waiting for 0.5~1hours until pressure and temperature of the standard vessel is stable. Take the N_2 pressure by hand as P1. Take by hand average reading of three PT1000s in the filling system as T_1. (here T_1 is compared T_{room} with range of $\pm 1^oC$);
8.	Close the SV1.5 valve, valve 3, and the N_2 bottle valve, disconnect SV1.5 from connector 3;
9.	Weight the SV1.5 with an electronic balance (0.1g in precision or better), as $M_{nitrogen1}$;
10.	Connect the filled SV1.5 back to the connector 3;
11.	Open valve 3, valve 1,vale 4 and the vacuum valve; open pressure regulator to pump the filling box and TTCS loop down to 30Pa;
12.	Close the vacuum valve, valve 1 and valve 4, and switch off the pump. Open the SV1.5 valve, then close SV1.5 valve, slowly turn on valve 1 to fill the N_2 into the TTCS loop to prevent pressure spike to the sensors. Repeating on and off SV1.5 valve and valve 1 in turn until pressure of TTCS loop is up to 1.5MPa, then open SV5.0 valve and valve 1 slowly at the same time.
13.	Wait a period of time (0.5~1hours), take by hand the loop pressure as P_2 and average reading of three PT1000s in the filling system as T_2.

	(here T_2 is compared T_{room} with range of $\pm 1^oC$); NOTE: Take by hand the highest and the lowest measurable temperature of the TTCS loop, the temperature difference should be less than 2^oC
14.	Close valve 1, valve 3, SV1.5 valve and the TTCS valve;
15.	Disconnect SV1.5 from the connector 3;
16.	Weight the SV1.5 with the electronic balance again, as $M_{nitrogen2}$;
17.	The nitrogen mass inside the loop is $M_{nitrogen}= M_{nitrogen1} - M_{nitrogen2} - M_{filling\ system}$, where
18.	The volume of the loop is determined by the ideal gas equation , where $m_{nitrogen}$= 28g/mol for nitrogen, and R is the gas constant;
19.	Determine the fill mass by $M_{C\_L}=V_{Loop} \cdot FR$ (where FR = 569.60 g/l).
20.	Calculate the filled mass for the standard vessel Set $\mathbf{M_{C\_VS}= M_{E\_VS} + M_{C\_L} + M_{C\_R}}$; NOTE: $\mathbf{M_{C\_R}} = \mathbf{M_{C\_R\_VS}} + \mathbf{M_{C\_R\_FT}} = V_{VS} \times \rho_G (T, P) + V_{FT} \times \rho_G (T, P)$ Here ρ_G (T, P) is determined by the chosen final temperature of the standard vessel and the final saturated pressure of the TTCS loop. **see appendix 8.5 For details.**

6.4 Fill a standard vessel with CO_2 from a Gas bottle

Table 6 Procedure of determination of filling a standard vessel with CO2 from a gas bottle

Step	Action
1.	Connect the SV1.5 (here anyone of the two SV1.5 can be used) to connector 3; **Note: Longer extend tube may be used, and height difference may exist between the SV1.5 and connector 3.** **The extend tube between connectors and valves must be mechanically supported to reduce strain.**
2.	Connect a high purity CO_2 gas bottle to connector 2;
3.	Close valve 1 ,valve 4 and the gas bottle valve, open valve 3, valve 2, pressure regulator, the vacuum valve and the SV1.5 valve;
4.	Open valve 3 until the pressure drop to 30Pa; Close the vacuum valve, open the bottle valve slowly, until the pressure up to 1MPa; Close the bottle valve and open the vacuum valve slowly, pump the SV1.5 until the pressure drop to 30Pa; Repeat such operations and flush with CO_2 twice.
5.	Place the vacuumed SV1.5 to a barrel with ice water mixture, switch off the pump;
6.	Open valve 2 and regulate the outlet pressure of gas bottle valve slowly until higher than 4MPa;
7.	After a period of time(2 hours~3 hours), close the SV1.5 valve, valve 2,valve 3 and the gas bottle valve; Estimate weight by electronic balance without disconnect, check whether it meets required mass in the SV1.5. If CO2 filled into the SV1.5 is less than the required mass, then put the SV1.5 to a barrel with ice water mixture again, open valve 2, valve 3 and the SV1.5valve. During the estimation of the weight without disconnection, the extend tube between the SV1.5 and filling box should be put up slightly in order to get a real weight of the filled SV1.5 as possible as we can, and even do that the extend tube still account for about 200g in total estimating weight.

8.	Disconnect SV1.5 from the filling box;
9.	Dry and clean the SV1.5 surface;
10.	Weight the filled SV1.5 with a electronic balance, as $\mathbf{M_{Filled\_VS}}$;
11.	Take by hand the filled CO_2 mass $\mathbf{M_{O\_VS} = M_{Filled\_VS} - M_{E\_VS}}$
12.	If $\mathbf{M_{O\_VS}}$ above what required ($\mathbf{M_{C\_L} + M_{C\_R}}$), open SV1.5 valve very slightly to release a little CO_2 gas, close SV1.5valve;
13.	Weight SV1.5again; repeats step 12 until m_{CO2} value meets the requirement ($\mathbf{M_{C\_L} + M_{C\_R}}$).
14.	Seal the extended tube and the filled SV1.5.

Note : According to previous experiments data, under 3.9MPa~4.2MPa filling mass rate of SV1.5 is about 300g～350g per hour near 0°C (mixture of ice and water)

6.5 Calibration of the filled mass

Table 7 Procedure of Calibration of the filled mass

Step	Action
1.	Set the filling environment temperature as 22°C
2.	Connect another standard vessel (**VS_B**) to connector 1 with the same extension filling tube for filling the TTCS loop; **<u>Note: Longer extend tube may be used, and height difference may exist between the TTCS loop and connector 1.</u>**

	The extend tube between connectors and valves must be mechanically supported to reduce strain.
3.	Connect a dry mechanical pump
4.	Close valve 2 and open the vacuum valve, valve 3, pressure regulator, valve 1, valve 4 and the **VS_B** valve;
5.	Pump **VS_B** until the pressure down to 30Pa;
6.	Connect the filled SV1.5 as the **VS_A** to connector 3;
7.	Wrap **VS_A** with heating tape and affix three PT1000 to the top, the middle, and the bottom of the **VS_A**, respectively.
8.	Open the vacuum valve slowly, and then valve 1, and valve 4 ;
9.	Pump the extension filling tube until the pressure down to 30Pa;
10.	Close the vacuum valve and valve 4;
11.	Open the **VS_A** valve slowly;
12.	After 5 minutes, switch on the filling box heater and the heating tape (to control the box temperature above 30°C);
13.	Watch the reading of the pressure sensor;
14.	When the pressure drops to ***6MPa***, close the **VS_B** valve, the pressure regulator, valve3, the **VS_A** valve, and valve 1, respectively;
15.	Switch off the filling box heater and the heating tape;
16.	unwrap **VS_A** ;
17.	Disconnect **VS_A** and **VS_B,** respectively;
18.	Weight **VS_A** and **VS_B**, as $\mathbf{M_{O\_R\_VSA}}$ and $\mathbf{M_{Filled\_VS\_B}}$, respectively
19.	From ($\mathbf{M_{Filled\_VS}}$– $\mathbf{M_{O\_R\_VS\_A}}$), we have the mass of CO_2 out of SV1.5; from ($\mathbf{M_{Filled\_VS\_B}}$ –$\mathbf{M_{E\_VS\_B}}$), we have the mass of CO_2 into **VS_B**;
20.	Obtain the mass loss M_{loss}= ($\mathbf{M_{Filled\_VS}}$– $\mathbf{M_{O\_R\_VS\_A}}$), – ($\mathbf{M_{Filled\_VSB}}$ –$\mathbf{M_{E\_VS\_B}}$),;

21.	Calculate the mass loss based on the filling tube volume $\mathbf{M_{C\_R\_FT}} = V_{FT} \times \rho_{CO2}(T)$;
22.	Obtain fill loss range of the filling tube by comparing $M_{\mathbf{loss}}$ and $\mathbf{M_{C\_R\_FT}}$ to determine performance of the current filling system
23.	Switch off the pump

6.6 Fill TTCS loop with CO_2 from SV1.5

Table 8 Procedure of filling TTCS loop with CO2 from SV1.5

Step	Action
1.	Set the filling environment temperature as 22^{o}C
2.	Connect the TTCS loop to connector 1 with an extended filling tube. **Note: Longer extend tube may be used, and height difference may exist between the TTCS loop and connector 1.** **The extend tube between connectors and valves must be mechanically supported to reduce strain.**
3.	**Connect a high purity CO_2 gas bottle to connector 2;**
4.	Put three PT1000 vertically by using adhesive tape in turn onto the side surface of the SV1.5, then wrap the SV1.5 with heater with Pt1000 in the joint of the heater. **Note: No.1 PT1000 for control, No.2 PT1000 and No.3 PT1000 for monitor.** **During the filling CO2 from the standard vessel to the TTCS loop, No.1 PT1000 should be paste on the middle of the standard vessel's side face; the other two PT1000s should be paste on the top and bottom of the standard vessel's side face.**
5.	Connect the filled SV1.5 to connector 3;

	<u>Note: Longer extend tube may be used, and height difference may exist between the SV1.5 and connector 3.</u> **<u>The extend tube between connectors and valves must be supported to reduce strain.</u>**
6.	Use the He-mass spectrometer to perform leak test before filling. If leak rate more than 5×10^{-5} ***mbar·l/s*** somewhere, reconnect it until the leak rate less than 5×10^{-5} ***mbar·l/s***
7.	Switch off the He-mass spectrometer and replace it with a dry mechanical pump.
8.	Insulate the filled vessel and connect the SV heater with socket in the filling box.
9.	Open the vacuum valve, valve 1, valve2, valve 3, valve 4 , the TTCS loop valve and pressure regulator
10.	Pump until pressure of the TTCS loop and the filling box down to 30Pa, close valve4, the vacuum valve and then open the gas bottle valve slowly to fill the loop with high purity CO_2 from the bottle, until the pressure up to 1MPa (to flush the loop)
11.	Close the gas bottle valve , open valve 1, valve 4 and the vacuum valve slowly;
12.	Pump the TTCS loop until the pressure down to 30Pa;
13.	repeat step 11 and 12 twice;
14.	Close valve 4, valve 2 and the vacuum valve; switch off the pump.
15.	Open the SV1.5 valve slowly, then close the SV1.5 valve, slowly open valve 1 to fill the CO2 into the TTCS loop to prevent pressure spike to the sensors. Repeating on and off the SV1.5 valve and valve 1 in turn until pressure of the TTCS loop is up to 3MPa, and then slowly open the SV1.5 valve and valve 1 in turn.
16.	Switch on the filling box heater and the standard vessel heater (to control the box temperature step by step to **30°C);**
17.	Using the small fans to cool the temperature of the accumulator to accelerate the filling
18.	Watch the reading of the digital pressure gauge;

19.	When the pressure drops down to ***5.995MPa***, close the TTCS loop valve, valve 1, the SV1.5 valve, and valve 3, respectively; **Note: here the pressure of 5.995MPa corresponds to CO_2 saturated temperature of 22^{o}C**
20.	Switch off the filling box heater and the standard vessel heater;
21.	unwrap the heating tape from the SV1.5;
22.	Disconnect the SV1.5 from the filling box;
23.	Weight the SV1.5 and compare it with ($\mathbf{M_{C\_R\_VS}} + \mathbf{M_{E\_VS}}$)
24.	After filling finished, the two extended filling tubes, the SV1.5 and the filling system must be carefully disconnect and seal.
25.	Carefully packing the two extended filling tubes, the SV1.5 and the filling system.

6.7 Venting CO_2 from the TTCS loop

Measures shall be taken to avoid damage or contamination arising from water condenses. Usually, below 15^{o}C, the condensing dropwise is visible. The ground accumulator heater (GAC in TTCE) is used to heat the accumulator.

The TTCE communication terminal is used to inspect the accumulator temperature while venting CO_2 from the TTCS loop. PT03 is taken as a detecting point. As soon as the temperature is below 18 ^{o}C, stop releasing operation.

A valve (TTCB valve) is connected to the filling port with a filter next to it. And then an exhaust stainless tube is connected to the filter and extended to the outside of the clean room. At the exit of the stainless tube, frost might be encountered because of throttle, thus, a hot air gun is also needed to suppress this phenomenon.

Monitor the loop pressure by TTCE terminal, stop venting until 1.5 bar.

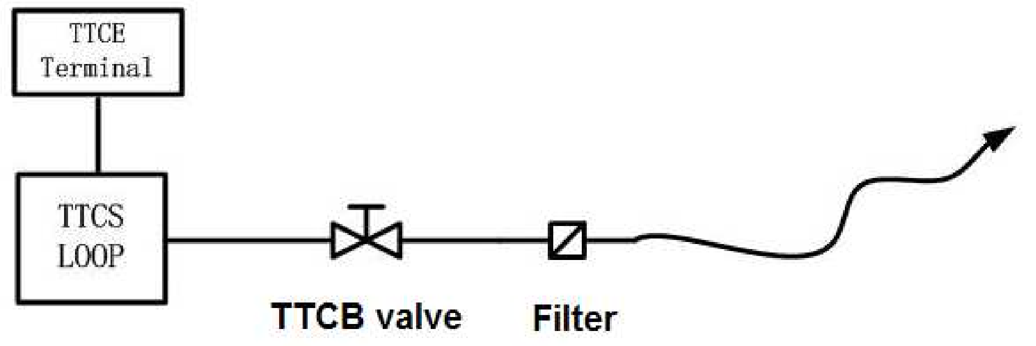

Figure 6-1 Schematic of venting CO_2 from TTCS loop

Table 9 Procedures for venting CO_2 from TTCS loop

1.	Switch on TTCE Terminal and go to the interface TTCE-2
2.	Write down the PT01 and PT03 temperatures
3.	Connect the TTCB valve, filter, stainless tube and extended tube
4.	Open the TTCB valve a little to release CO_2 gas slowly while switch on the ground accumulator heater (GAC) with 30% Monitor the PT01 and PT 03, once one temperature is below 18 ^oC, stop venting.
5.	Use a hot air gun to heat up the exit of the stainless tube
6.	Repeat steps 0 and 0, until the pressure is down to 1.5 bar
7.	Check the reading of the TTCB' APS. Close the TTCB valve when the pressure is low down 1.5 Bar.

8.	Switch off TTCE Terminal

6.8 Temporary sealing the TTCS loop (FM)

1. Close the fill valve to for further sealing the TTCS FM loop;
2. Clap the tube to the TTCS loop.

Note:
The volume of the filling tube from the box to the TTCS loop may differ from that to SV1.5. In this case, an estimation of this tube volume can be made: Taking a filling tube of one meter in length, 3mm in inner diameter as an example, the volume is 14 ml. The pressure is 6MPa at the filling temperature of 32°C is chosen as final thermodynamics state. Therefore, the mass loss in the filling is about 0.78g/m. This should be taken into account for the calculation of m_{loss}.

7. FILLING PROCEDURE DATA SHEET

7.1 Preparation procedures

Preparation procedures						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions					
2.	Temperature: □ / Humidity: %RH / Pressure: MPa					
3.	Calibrate the volume of the filling box;(Vbox= 0.021 liter)					
4.	Calculate the volume of the two connecting tubes and valves;					
5.	Select filling environmental temperature, e.g., 22°C;	T_{room}				
6.	Wrap a heating tape around the filling tube outside the box;					
7.	Prepare an electronic balance with full scale more than 4200g and accuracy of 0.1g;					
8.	Prepare a bottle of pure N_2 gas for volume calibration					
9.	Prepare high purity (99.99%)CO_2 bottle for filling;					
10.	Prepare a barrel with ice water mixture for cooling the vessel;					
11.	Prepare a fan for cooling TTCS accumulator during filling;					
12.	Connect a dry mechanical pump to the vacuum connector;					

7.2 Filling system leak rate examination

Filling system leak rate examination						
Date:	**Company:**		**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions					
2.	Temperature: □ / Humidity: %RH / Pressure: MPa					
3.	Connect the two SV 1.5s to connector 1 and connector 3, respectively. Close valve 2.					
4.	Measure the background leak rate of the “He mass spectrometer”		$<5\times10^{-10}$ ***mbar·l/s***.			
5.	Connect the He mass spectrometer to the vacuum connector					
6.	Open valve1, valve 3 and valve 4 Vacuum the filling system					
7.	Measure the leak rate of the filling system without helium gas.		$<5\times10^{-8}$ ***mbar·l/s***.			
8.	Using negative method, blowing helium gas, to measure the leak rate of these connectors, valves. Leak rate for each connector should be below 5×10^{-5} ***mbar·l/s***.		$<5\times10^{-5}$ ***mbar·l/s***.			
9.	Disconnect the He mass spectrometer and measure the background leak rate of the equipment.		$<5\times10^{-10}$ ***mbar·l/s***.			

Filling system leak rate examination						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
10.	**Flash the filling system**					
11.	Connect the N_2 bottle to the connector 2					
12.	Open the bottle valve and valve 2 Pressurize the filling system slowly		40±1bar			
13.	Close valve 2 and open vacuum valve Release the N2 from the system slowly		2±0.5bar			
14.	Open the bottle valve and valve 2 Pressurize the filling system slowly		40±1bar			
15.	Close valve 2 and open vacuum valve Release the N2 from the system slowly		2±0.5bar			
16.	Open the bottle valve and valve 2 Pressurize the filling system slowly		40±1bar			
17.	Close valve 2 and open vacuum valve Release the N2 from the system slowly		2±0.5bar			

7.3 Determination of filling system volume

Filling system volume determination								
Date:		Company:		Location:		Engineer:		
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions							
2.	Temperature: □	Humidity: %RH	Pressure: MPa					
3.	Affix any two of the three PT1000s on the side surface of the SV1.5. and leave the rest one to monitor environmental temperature.							
4.	Connect the SV1.5 (any of the three stand vessels can be used) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **Note: The extend tube between connectors and valves must be supported to reduce strain.** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)							
5.	Connect the N_2 bottle to connector 2;							
6.	Connect a dry mechanical pump to vacuum connector							

Filling system volume determination						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
7.	seal the extend filling tube with the other end connected to connector 1 of the filling box; Note: the need of redetermination of the filling system is due to the use of additional extend filling tubes, therefore, the extend filling must be connected to the filling box with the other end sealed. On the other hand, we could skip this procedure if we can rely on the calculation of the volume of the additional extend filling tubes. Then : $V_{FT}=V_{filling\_system}+V_{C\_extend\ tubes}$		s			
8.	Close valve 1, valve 4 and the N_2 bottle valve, open valve 2, valve 3, the SV1.5 valve, pressure regulator and the vacuum valve slowly, pump the SV1.5 and the filling box down to 30Pa;	Evacuate Time Vacuum gauge reading	1~2 hours ≤30Pa			
9.	Close the vacuum valve open the valve of the N_2 bottle					

Filling system volume determination						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
10.	Fill the SV1.5 with N_2 up to 4.5MPa, then close valve 2; wait until pressure and temperature of the standard vessel is stable. Take by hand the pressure as **P1'.** Take by hand the average reading of the three PT1000s in the filling system as T_1'. (here T_1' is comparable to T_{room} with range of ±1°C);	Evacuate Time **P1'** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	0.5~1hours T_{room}±1°C T_{room}±1°C T_{room}±1°C			
11.	Close the filled SV1.5 valve, valve 3, and the N_2 bottle valve, disconnect the filled SV1.5 from connector 3;					
12.	Weight the filled SV1.5 with an electronic balance (0.1g precision or better), as $M'_{nitrogen1}$;					
13.	Connect the filled SV1.5 back to the connector 3;					
14.	Open valve 3, valve 1,vale 4 and the vacuum valve; open pressure regulator to pump the filling box down to 30Pa;					
15.	Close the vacuum valve, valve 1 and valve 4, switch off the pump. Open the SV1.5 valve and close SV1.5 valve, slowly open valve 1 to fill the N_2 into the filling box and tubes.	DPS reading of the TTCS loop	≤0.5Bar			

Filling system volume determination						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
16.	Wait about ten mins, take by hand the pressure as **P2’** and the average reading of the three PT1000s in the filling system as T_2’. (here T_2’ is comparable to T_{room} with range of ±1°C);	**P2’** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	 T_{room}±1°C T_{room}±1°C T_{room}±1°C			
17.	Close valve 1, valve 3, SV1.5 valve;					
18.	Disconnect SV1.5 from the connector 3;					
19.	Weight the SV1.5 with the electronic balance again, as $M'_{nitrogen2}$;					
20.	The nitrogen mass inside the filling system including tubes is: $M_{FT}= M'_{nitrogen1} - M'_{nitrogen2}$,					
21.	The volume of the filling system including tubes is determined by the ideal gas equation , where $V_{FT} = \frac{M_{FT} R T_2'}{m_{nitrogen} P_2'}$ $m_{nitrogen}$= 28g/mol for nitrogen, and R is the gas constant;					

7.4 Determination of TTCS volume

TTCS volume determination									
Date:			**Company:**		**Location:**		**Engineer:**		
Step No.	**Action and Description**				**Monitoring**	**Value**	**Result**	**Comment**	**Time**
22.	Take by hand environment conditions								
23.	Temperature: □	Humidity: %RH	Pressure: MPa						
24.	Affix any two of the three PT1000s on the side surface of the SV1.5. and leave the rest one to monitor environmental temperature.								
25.	Connect the SV1.5 (any of the three stand vessels can be used) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **Note: The extend tube between connectors and valves must be supported to reduce strain.** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)								
26.	Connect the N_2 bottle to connector 2;								

TTCS volume determination						
Date:		**Company:**		**Location:**	**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
27.	Connect a dry mechanical pump to vacuum connector					
28.	Connect the TTCS loop to connector 1; Longer extend tubes may be used and height difference may exist between the TTCS loop and connector 1. **Note: The extend tube between connectors and valves must be supported to reduce strain.**					
29.	Close valve 1, valve 4 and the N_2 bottle valve, open valve 2, valve 3, the SV1.5 valve, pressure regulator and the vacuum valve slowly, pump the SV1.5 and the filling box down to 30Pa;	Evacuate Time Vacuum gauge reading	1~2 hours ≤30Pa			
30.	Close the vacuum valve open the valve of the N_2 bottle					
31.	Fill the SV1.5 with N_2 up to 4.5MPa, then close valve 2; waiting until pressure and temperature of the standard vessel is stable. Take by hand the pressure as **P1.** Take by hand the average reading of the three PT1000s in the filling system as T_1. (here T_1 is comparable to T_{room} with range of $\pm 1^oC$);	Evacuate Time **P1** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	0.5~1hours $T_{room}\pm 1^oC$ $T_{room}\pm 1^oC$ $T_{room}\pm 1^oC$			

TTCS volume determination						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
32.	Close the filled SV1.5 valve, valve 3, and the N_2 bottle valve, disconnect the filled SV1.5 from connector 3;					
33.	Weight the filled SV1.5 with an electronic balance (0.1g precision or better), as $M_{nitrogen1}$;					
34.	Connect the filled SV1.5 back to the connector 3;					
35.	Open valve 3, valve 1,vale 4 and the vacuum valve; open pressure regulator to pump the filling box and TTCS loop down to 30Pa;					
36.	Close the vacuum valve, valve 1 and valve 4, switch off the pump. Open the SV1.5 valve and close SV1.5 valve, slowly open valve 1 to fill the N_2 into the TTCS loop to prevent pressure spike to the sensors. Repeating on and off the SV1.5 valve and valve 1 in turn until pressure of the TTCS loop is up 1.5MPa, and then slowly open the SV1.5 valve and valve.	DPS reading of the TTCS loop	≤0.5Bar			

TTCS volume determination						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
37.	Wait a period of time (0.5~1hours), take by hand the loop pressure as **P2** and the average reading of the three PT1000s in the filling system as T_2. (here T_2 is comparable to T_{room} with range of ±1°C);	**P2** No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	 T_{room}±1°C T_{room}±1°C T_{room}±1°C			
38.	Close valve 1, valve 3, SV1.5 valve and the TTCS valve;					
39.	Disconnect SV1.5 from the connector 3;					
40.	Weight the SV1.5 with the electronic balance again, as $M_{nitrogen2}$;					
41.	The nitrogen mass inside the loop and the filling system is: $M_{nitrogen}= M_{nitrogen1} - M_{nitrogen2}$,					
42.	The volume of the loop is determined by the ideal gas equation , where $$V_{Loop} = \frac{M_{\mathrm{nitrogen}} R T_2}{m_{\mathrm{nitrogen}} P_2} - V_{FT}$$ $m_{nitrogen}$= 28g/mol for nitrogen, and R is the gas constant;					
43.	Determine the fill mass by $\mathbf{M_{C_L}} = V_{Loop}$FR (where FR = 569.60 g/l).					

TTCS volume determination						
Date:	Company:	Location:		Engineer:		
Step No.	Action and Description	Monitoring	Value	Result	Comment	Time
44.	Calculate the filled mass for the standard vessel Set $\mathbf{M_{C\_VS}} = \mathbf{M_{E\_VS}} + \mathbf{M_{C\_L}} + \mathbf{M_{C\_R}}$; NOTE: $\mathbf{M_{C\_R}} = \mathbf{M_{C\_R\_VS}} + \mathbf{M_{C\_R\_FT}} = V_{VS} \times \rho_G (T, P) + V_{FT} \times \rho_G (T, P)$ Here ρ_G (T, P) is determined by the chosen final temperature of the standard vessel and the final saturated pressure of the TTCS loop.					
45.	Repeat the above procedure of TTCS volume determination again for confirmation					

7.5 Fill a standard vessel with CO_2 from a Gas bottle

Fill a standard vessel with CO_2 from a Gas bottle								
Date:		Company:	Location:		Engineer:			
Step No.	Action and Description			Monitoring	Value	Result	Comment	Time
1.	Take by hand environment conditions							
2.	Temperature: □	Humidity: %RH	Pressure: MPa					

Fill a standard vessel with CO_2 from a Gas bottle							
Date:		**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**		**Monitoring**	**Value**	**Result**	**Comment**	**Time**
3.	Connect the SV1.5 (here anyone of the two SV1.5 can be used) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **<u>Note: The extend tube between connectors and valves must be supported to reduce strain.</u>** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)						
4.	Connect a high purity CO_2 gas bottle to connector 2;						
5.	Connect a dry mechanical pump to vacuum connector						
6.	Close valve 1 ,valve 4 and the gas bottle valve, open valve 3, valve 2 , pressure regulator, the vacuum valve and the SV1.5 valve;						

Fill a standard vessel with CO_2 from a Gas bottle						
Date:	**Company:**		**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
7.	Open valve 3 until the pressure drop to 30Pa; Close the vacuum valve, open the bottle valve slowly, until the pressure up to 1MPa; Close the bottle valve and open the vacuum valve slowly, pump the SV1.5 until the pressure drop to 30Pa; **Repeat such operations and flush with CO_2 twice (totally three times).**	Evacuate Time Vacuum gauge reading	1~2 hours ≤30Pa ≤30Pa ≤30Pa			
8.	Place the vacuumed SV1.5 to a barrel with ice water mixture, switch off the pump;					
9.	Open valve 2 and regulate outlet pressure of gas bottle valve slowly until higher than 4MPa;	Outlet pressure of gas bottle	≥ 4MPa			

Fill a standard vessel with CO_2 from a Gas bottle						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
10.	After a period of time (2 hours~3 hours), close the SV1.5 valve, valve 2 and valve 3; Estimate weight by electronic balance without disconnecting SV1.5, check whether it meets required mass in the SV1.5. If CO2 filled into the SV1.5 is less than the required mass, then put the SV1.5 back to the barrel, and open valve 2, valve 3 and the SV1.5valve. During the estimation of weight without disconnecting SV1.5, the extend tube between the SV1.5 and filling box should be put up slightly in order to get real weight of the filled SV1.5 as possible as we can, and even though, the extend tube still account for about 200g in total estimating weight.	Waiting time Estimating weight	2 hours~3 hours $\geqq \mathbf{M_{C\_VS}+200g}$			
11.	Disconnect the SV1.5 from the filling box, and dry the SV1.5 surface; Seal the extended tube.	Dry and clean the SV1.5 surface				
12.	Weight the filled SV1.5 with a electronic balance, as $\mathbf{M_{Filled\_VS}}$;	$\mathbf{M_{Filled\_VS}}$				
13.	Take by hand the filled CO_2 mass $\mathbf{M_{O\_VS} = M_{Filled\_VS} - M_{E\_VS}}$;	$\mathbf{M_{O\_VS}}$				
14.	If $\mathbf{M_{O\_VS}} > \mathbf{(M_{C\_VS} - M_{E\_VS})}$, open the SV1.5 valve very slightly to release a little CO_2 gas, close SV1.5 valve;					

Fill a standard vessel with CO_2 from a Gas bottle						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
15.	Weight the SV1.5 again; repeats step 12 to 14 until $\mathbf{M_{O\_VS}}$ value meets the requirement **(=$\mathbf{M_{C\_VS} - M_{E\_VS}}$)**.	$\mathbf{M_{O\_VS}}$	**($\mathbf{M_{C\_VS} - M_{E\_VS}}$) ±1g**			
16.	Seal the filled SV1.5 valve for cleanness					

7.6 Calibration of the filled mass

Calibration of the filled mass								
Date:		**Company:**		**Location:**		**Engineer:**		
Step No.	**Action and Description**			**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions							
2.	Temperature: □	Humidity: %RH	Pressure: MPa					
3.	Set the filling environment temperature as 22°C							
4.	Connect the filled SV1.5 (the $SV1.5_{new1}$) to connector 3; Note: Longer extend tube may be used and height difference may exist between the SV1.5 and connector 3. **Note: The extend tube between connectors and valves must be supported to reduce strain.** (For the new connectors, wrench screw nuts with one and quarter circles. For the used connectors, wrench the screw nuts further tightly.)							
5.	Connect another standard vessel (**VS_B**) to connector 1 with the same extension filling tube for filling the TTCS loop;							
6.	Connect a dry mechanical pump to vacuum connector							

Calibration of the filled mass						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
7.	Close valve 2 and open the vacuum valve, valve 3, pressure regulator, valve 1, valve 4 and the VS_B valve;					
8.	Pump the VS_B until the pressure down to 30Pa;	Vacuum gauge reading	≤30Pa			
9.	Close the vacuum valve and valve 4;					
10.	Open the VS_A valve slowly;					
11.	Switch on the filling box heater with and the standard vessel heater (to control the box temperature to 25^{o}C step by step); If possible, the standard vessel heater should increase the temperature with an interval of 5~10^{o}C. If heat power of the filling box heater is not enough, using additional heat gun to warm up the **VS_A**	No.1 Pt1000 No.2 Pt1000 No.3 Pt1000	≤30^{o}C ≤30^{o}C ≤30^{o}C			
12.	Using the fans to cool the **VS_B**					

Calibration of the filled mass						
Date:		Company:	Location:		Engineer:	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
13.	Watch the reading of the digital pressure gauge. When the pressure and temperature is close to the set value, switch on the heating tape on the filling tubes. When the pressure is equal to **5.995MPa**, close the TTCS loop valve, valve 1, the SV1.5 valve, and valve 3, respectively;	APS reading of the filling system Temperatures of the filling box and tubes	*5.995±0.1MPa* *25°C*			
14.	Switch off the filling box heater and the heating tape;					
15.	Unwrap the **VS_A**;					
16.	Disconnect the **VS_A** and the **VS_B** r espectively;					
17.	Weight the **VS_A** and the **VS_B**, as $\mathbf{M_{O\_R\_VSA}}$ and $\mathbf{M_{Filled\_VS\_B}}$, respectively					
18.	From ($\mathbf{M_{Filled\_VS}}$– $\mathbf{M_{O\_R\_VS\_A}}$), we have the mass of CO_2 out of SV1.5; from ($\mathbf{M_{Filled\_VS\_B}}$ –$\mathbf{M_{E\_VS\_B}}$), we have the mass of CO_2 into **VS_B**;	($\mathbf{M_{Filled\_VS}}$– $\mathbf{M_{O\_R\_VS\_A}}$) ($\mathbf{M_{Filled\_VS\_B}}$ –$\mathbf{M_{E\_VS\_B}}$)				
19.	Obtain the mass loss M_{loss}=($\mathbf{M_{Filled\_VS}}$–$\mathbf{M_{O\_R\_VS\_A}}$)–($\mathbf{M_{Filled\_VSB}}$ –$\mathbf{M_{E\_VS\_B}}$);	M_{loss}				

Calibration of the filled mass						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
20.	Calculate the mass loss based on the filling tube volume $\mathbf{M_{C\_R\_FT}} = V_{FT} \times \rho_G(T)$; where $T=25^{o}C$	$\mathbf{M_{C\_R\_FT}}$				
21.	Obtain fill loss range of the filling tube by comparing $M_{\mathbf{loss}}$ and $\mathbf{M_{C\_R\_FT}}$ to determine performance of the current filling system					
22.	Switch off the pump.					
23.	Seal the filled SV1.5 valve for cleanness					

7.7 Fill TTCS loop with CO_2 from SV1.5

Fill TTCS loop with CO_2 from SV1.5							
Date:		**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**		**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions						
2.	Temperature: □	Humidity: %RH Pressure: MPa					
3.	Set the filling environment temperature as 22^oC						
4.	Connect the TTCS loop to connector 1 with an extended filling tube. **Note: Longer extend tubes may be used and height difference may exist between the TTCS loop and connector 1.** **The extend tube between connectors and valves must be mechanically supported to reduce strain.**						
5.	**Connect a high purity CO_2 gas bottle to connector 2;**						

Fill TTCS loop with CO_2 from SV1.5						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
6.	Put three PT1000 vertically by using adhesive tape in turn on the side surface of the SV1.5, then wrap the SV1.5 with heater with Pt1000 in the joint of the heater. **(Note: No.1 PT1000 for control, No.2 PT1000 and No.3 PT1000 for monitor.** **During filling CO_2 from the standard vessel to the TTCS loop, No.1 PT1000 should be paste on the middle of the standard vessel's side face; the other two PT1000s should be pasted on the top and bottom of the standard vessel's side face).**					
7.	Connect the filled SV1.5 to connector 3; **Note: Longer extend tubes may be used and height difference may exist between the TTCS loop and connector 1.** **The extend tube between connectors and valves must be mechanically supported to reduce strain.**					
8.	Use the He-mass spectrometer to perform leak test before filling. If leak rate more than 5×10^{-5} $mbar\cdot l/s$ somewhere, reconnect it until the leak rate is less than 5×10^{-5} $mbar\cdot l/s$	LR	$<5\times10^{-5}$ $mbar\cdot l/s$			

Fill TTCS loop with CO_2 from SV1.5						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
9.	Stop the He-mass spectrometer and replace it with a dry mechanical pump.					
10.	Follow the section of 7.5 (**fill a standard vessel with CO_2 to from a Gas bottle**) to fill SV1.5 to $\mathbf{M_{filled\_SV}}$;	$\mathbf{M_{filled\_SV}}$				
11.	Put three PT1000 vertically on the side surface of the SV1.5 by using adhesive tape. Then wrap the SV1.5 with heater with Pt1000 in the joint of the heater. **Note: check them can work or not** (No.1 PT1000 for control, No.2 PT1000 and No.3PT1000 for monitor. During the filling CO2 from the standard vessel to the TTCS loop No.1 PT1000 should be paste on the middle of the standard vessel's side face; the other two PT1000s should be paste on the top and bottom of the standard vessel's side face).	NO.1 Pt1000 reading NO.2 Pt1000 reading NO.3 Pt1000 reading	Ok or not Ok or not Ok or not			
12.	Insulate the filled vessel and connect the SV heater with the socket in the filling box.					
13.	Open the vacuum valve, valve 1, valve2, valve 3, valve 4 , the TTCS loop valve and the pressure regulator					

Fill TTCS loop with CO_2 from SV1.5							
Date:		**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**	
14.	Wait until pressure of the TTCS loop, the filling box and two extend tubes down to 30Pa or lower. Close valve4, the vacuum valve, and valve 1. then open the gas bottle valve slowly and valve 1 to fill the loop with high purity CO_2 from the gas bottle, until the pressure up to 1MPa (flush the loop)	Evacuate Time Vacuum gauge reading	1~2hours ≤30Pa ≥1MPa				
15.	Close the gas bottle valve , open valve 1, valve 4 and the vacuum valve slowly;						
16.	Pump the TTCS loop, the filling box and two extend tubes until the pressure down to 30Pa or lower; Close valve4, the vacuum valve, and valve 1. then open the gas bottle valve slowly and valve 1 to fill the loop with high purity CO_2 from the gas bottle, until the pressure up to 1MPa (flush the loop)	Evacuate Time Vacuum gauge reading	0.6 hour ≤30Pa ≥1MPa				
17.	Pump the TTCS loop, the filling box and two extend tubes until the pressure down to 30Pa or lower;	Evacuate Time Vacuum gauge reading	0.6 hour ≤30Pa				
18.	Close valve 4, the vacuum valve; switch off the pump.						

Fill TTCS loop with CO_2 from SV1.5						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
19.	Put the SV1.5 with the connected tube on the electronic balance. Open the SV1.5 valve slowly, then close the SV1.5 valve, slowly open valve 1 to fill the CO2 into the TTCS loop to prevent pressure spike to the sensors. Repeating on and off the SV1.5 valve and valve 1 in turn until pressure of the TTCS loop is up to 3MPa, and then slowly open the SV1.5 valve and valve 1.	DPS reading of the TTCS loop	≤0.5bar			
20.	Switch on the filling box heater and the standard vessel heater (to control the box temperature to 25°C step by step); If possible, the standard vessel should be heated with a temperature interval of 5~10°C. If heat power of the filling box heater is not enough, using additional heat gun to warm up the SV1.5	NO.1 Pt1000 reading NO.2 Pt1000 reading NO.3 Pt1000 reading	*30±0.5°C* *30±1°C* *30±1°C*			
21.	Use the fan to cool the accumulator					

Fill TTCS loop with CO_2 from SV1.5						
Date:	Company:	Location:		Engineer:		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
22.	Watch the reading of the digital pressure gauge. When the pressure and temperature is close to the set-value, switch on the heating tape of the filling tubes. When the pressure is equal to **5.995MPa**, close the TTCS loop valve, valve 1, the SV1.5 valve, and valve 3, respectively;	Waiting time APS reading of the TTCS loop (P_{TTCB}) APS reading of the filling system ($P_{filling\ system}$)	3~4 hours 5.995±0.1MPa 5.995±0.1MPa			
23.	Switch off the filling box heater and the standard vessel heater;					
24.	Unwrap the heating tape from the SV1.5;					
25.	Disconnect the SV1.5 from the filling box, clean surface of the SV1.5					
26.	Weight the SV1.5 as ($\mathbf{M_{O\_R\_VS}+ M_{E\_VS}}$), compared ($\mathbf{M_{O\_R\_VS}+ M_{E\_VS}}$) with ($\mathbf{M_{C\_R\_VS} + M_{E\_VS}}$)	($\mathbf{M_{O\_R\_VS}+M_{E\_VS}}$)	($\mathbf{M_{C\_R\_VS}+M_{E\_VS}}$) ±5g			
27.	Filling finished.					
28.	After the filling is finished, the two extended filling tubes, the SV1.5 valve and the filling system must be carefully disconnected and sealed.					
29.	Carefully packing the two extended filling tubes, the SV1.5 and the filling system.					

7.8 Venting CO_2 from the TTCS loop

Venting CO2 from the TTCS loop						
Date:		**Company:**	**Location:**		**Engineer:**	
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
1.	Take by hand environment conditions					
2.	Temperature: ℃	Humidity: %RH	Pressure: MPa					
3.	Switch on TTCE Terminal and go to the interface TTCE-2					
4.	Write down the PT01 and PT03 temperatures	PT01 and PT03	>22°C			
5.	Connect the TTCB valve, filter, stainless tube and extended tube					
6.	Open the TTCB valve a little to release CO_2 gas slowly while switch on the ground accumulator heater (GAC) with 30% Monitor PT01 and PT 03, once either temperature is below 18 °C, stop venting by closing TTCB valve.	PT01 and PT03	>17°C			
7.	Use a hot air gun, or hot water bath, to heat up the exit of the stainless exhaust tube					
8.	Repeat steps 6 and 7, until the pressure is down to 1.5 bar	APS reading	1.5±0.1bar			

Venting CO2 from the TTCS loop						
Date:	**Company:**	**Location:**		**Engineer:**		
Step No.	**Action and Description**	**Monitoring**	**Value**	**Result**	**Comment**	**Time**
9.	Close the TTCB valve.	APS reading	1.5±0.1bar			
10.	Switch off TTCE Terminal					

8. APPENDIX

8.1 Error analysis of volume determination of a vessel by weighting the gas mass inside

Assumption: no leakage of measuring system, including the container, during the period of measurement; the electronic balance employed has the best accuracy of 0.1g; and the temperature be controlled within 0.5K.

According to the ideal gas equation:

$$PV = \frac{M}{m} RT \qquad \text{(A1.1)}$$

Where m is the mole mass of the gas in use (in g/mole), M is the net gas mass to be measured, and R is the gas constant, T is temperature of 22^{o}C in thermal bath respectively. For nitrogen, m=28g/mole, and a vessel of about 1.5liters, M≈76.3g. From (A1.1), we have

$$\frac{\Delta V}{V} = \frac{\Delta P}{P} + \frac{\Delta M}{M} + \frac{\Delta T}{T} = \frac{\Delta P}{P} + 2\frac{\Delta M_{\mathrm{B}}}{M} + \frac{\Delta T}{T} \qquad \text{(A1.2)}$$

As ΔP is limited by the pressure sensor, with the pressure sensor available with accuracy of ±0.2% FS, by setting P to the FS of the pressure sensor, (e.g., 10MPa), we have the lowest error of 0.44% for $\Delta P/P$. For using nitrogen as an ideal gas, $\Delta V/V$ is about 0.89 %.

Gas used	N_2
m (g/mole)	28
M(g)	76.3
ΔM/M(%)	0.26
ΔT/T(%)	0.17
ΔP/P(%)	0.44
ΔV/V(%)	0.89

8.2 Error analysis of volume determination of TTCS loop by indirectly measuring the Nitrogen mass inside the loop

Assumption: no leakage to the measuring system, including the TTCS loop and the filling system, during the filling; and the temperature and its uniformity be controlled within ±2K; the volumes of the standard vessel to fill to loop (V_{VS}) is about 1.5 liter, and the volume of the loop is about 1.5 liter ($V_{loop} \approx V_{VS}$); the accuracy of the electronic balance is 0.1g (ΔM_B); no nitrogen absorption on the inner surface of the TTCS loop that made of stainless steel.

1) Error analysis for the measured volume the filling system including the filling tubes.

Fill nitrogen with the SV1.5 to a pressure of 4.5MPa; weight the mass of the filled SV1.5 $M'_{nitrogen1}$. Connect the vessel to the filling system and release the nitrogen slowly. Weight the mass of the SV1.5 again $M'_{nitrogen2}$, the nitrogen mass filled into the filling system can then be calculated: $M_{FT} = M'_{nitrogen1} - M'_{nitrogen2}$, and $\Delta M_{nitrogen} = \Delta M_1 + \Delta M_2 \approx 2\Delta M_B$. To the volume of the filling tube of the filling system is about 0.042 liter. Even with additional tube of 5m long and 3mm in inner diameter, which is about 0.035 liter, the nitrogen mass inside filling tube is about a few percentages of that in the SV1.5 and the pressure inside is about the same as the initial pressure of SV1.5. The estimated N_2 mass is:

$$M_{FT} \approx \frac{1}{20}\frac{P_0 V_{VS} m_{nitrogen}}{RT_0};$$

Assuming $P_{FT} \approx P_0$, the error of V_{FT}

$$\frac{\Delta V_{FT}}{V_{FT}} = \frac{\Delta P_{FT}}{P_{FT}} + 2\frac{\Delta M_B}{M_{FT}} + \frac{\Delta T_{FT}}{T_{FT}}$$

$$= \frac{0.1\%FS}{P_0} + 40\frac{\Delta M_B RT_0}{P_0 V_{VS} m_{nitrogen}} + \frac{\Delta T_{FT}}{T_{FT}}$$

is about 6.3%

2) Error analysis for the measured volume of the loop and filling tube

Similarly, connect the filled vessel to a vacuumed loop and release the nitrogen inside the filled VS1.5 slowly until equilibrium is established. Weight the mass of the SV1.5 again $M_{nitrogen2}$, the nitrogen mass filled into the loop can then be calculated: $M_{loop+FT} = M_{nitrogen1} – M_{nitrogen2}$, and $\Delta M_{loop+FT} = \Delta M_1 + \Delta M_2 \approx 2\Delta M_B$. As V_{VS} is about half of the total volume, the nitrogen mass inside the loop and filling tube is about half of the initial filled mass of nitrogen in the SV1.5, and the pressure inside the loop and filling tube is about half of the initial pressure of SV1.5.

$$M_{loop+FT} = \frac{1}{2}\frac{P_0 V_{\mathrm{VS}} m_{nitrogen}}{RT_0} \quad \text{And } P_1 \approx \frac{1}{2}P_0;$$

Assuming $T_0 = T_{loop}$, and ΔT_{loop} is about ±2K

The error of $V_{\mathrm{loop+FT}}$:

$$\frac{\Delta V_{\mathrm{loop+FT}}}{V_{loop+FT}} = \frac{\Delta P_1}{P_1} + 2\frac{\Delta M_{\mathrm{B}}}{M_{loop+FT}} + \frac{\Delta T_{loop}}{T_{loop}}$$

$$= 2\frac{0.2\%FS}{P_0} + 4\frac{\Delta M_B RT_0}{P_0 V_{\mathrm{VS}} m_{nitrogen}} + \frac{\Delta T_{loop}}{T_{loop}}$$

is about 2.1%.

3) Error analysis for the measured volume of the loop

The error of V_{loop}:

$$\frac{\Delta \mathrm{V}_{\mathrm{loop}}}{\mathrm{V}_{\mathrm{loop}}} = \frac{2.1\% \times 1.5 + 6.3\% \times (0.042 + 0.035)}{1.5}$$

is about 2.4%

In the assumption, no absorption of nitrogen is considered. This is achieved by performing the measurement for at least twice. Let the nitrogen absorption, if there is any, reach its saturated value during the first measurement, so for the following measurement, there should not be additional nitrogen absorption happen.

8.3 Error analysis of the total filled mass of TTCS loop

Assumption: There is no leakage for the filling system, and the TTCS loop during the period of filling and measurement; the temperature is controlled within ±2.0K for filling procedure without thermal bath; and the volume of the loop is about 1.5 liter; the electronic balance employed has the best accuracy of ±0.1g (ΔM_B). (Based on our experiments), any mass loss is less than 1g.

According to Annex 8.2, the error of the loop volume is about 2.4%, so the calculated filled mass $m_{cal} = FrV_{loop}$ (≈860g) has also the error of=2.4%.

(1) The error from weighting CO_2 mass is

$\Delta M=\Delta M_2-\Delta M_1=2\ \Delta M_B=\pm 0.2g$.

(2) The error from the filling loss in the filling system, the CO_2 mass remained the filling tube of filling system plus additional tube with length of 5m (0.077L) is about 12.75g based on NIST refrigerant properties database.

The error of total filled mass of TTCS is

$$\frac{\Delta M_{\mathrm{CO2\_loop}}}{\mathrm{M}_{\mathrm{CO2\_loop}}} = \frac{\Delta \mathrm{V}_{\mathrm{loop}}}{\mathrm{V}_{\mathrm{loop}}} + \frac{2M_B + M_{loos}}{\mathrm{V}_{\mathrm{loop}} \times \mathrm{FR}} \approx 2.4\% + 1.2/1.5/569.6 \approx 2.6\%$$

Therefore, we have total CO_2 mass accuracy of ±2.6%, which meets the requirement of ±4%.

Conclusion:

Although the volume error is larger than 2% (required in Ref.1), the mass error (2.6%) is still within that required (4% in the Ref.1)

The impact of using electronic balance with accuracy of 0.1g other than 0.01g is that, the error of loop volume determination is obviously increased, which will be transferred to corresponding mass error. The impact on weighting itself is little.

8.4 Calculation of the required filling mass into the SV1.5

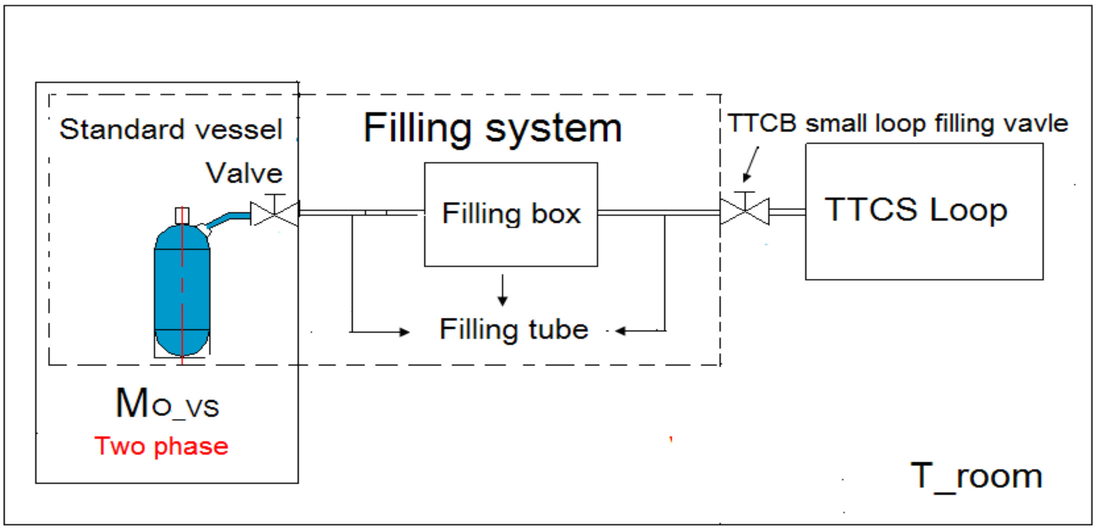

Figure 8-1 CO2 initial state and mass distribution between the filling system and the TTCS loop before starting filling

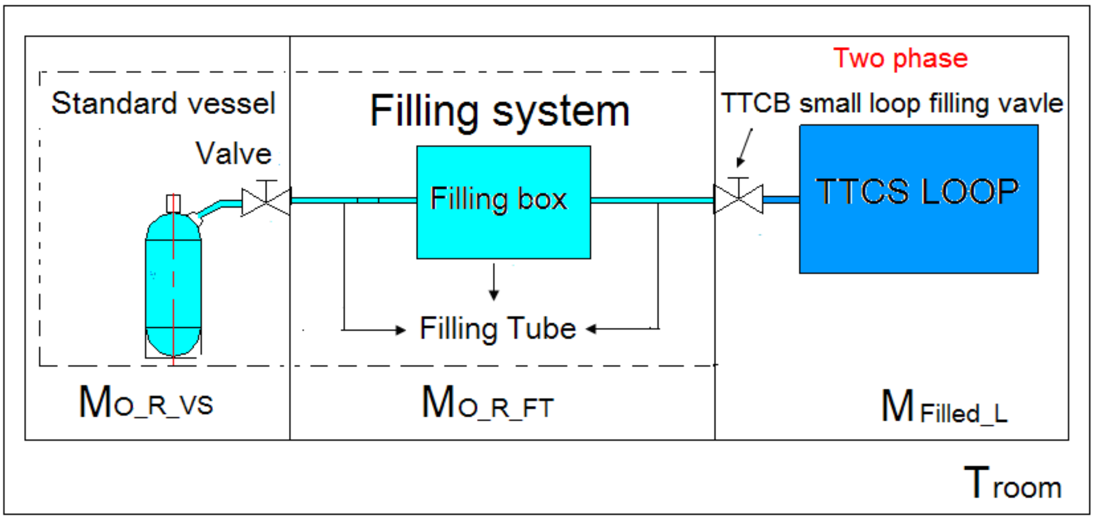

Figure 8-2 CO_2 finial state and mass distribution between the filling system and the TTCS loop after finishing filling

It can be concluded from Figure 8-1 and Figure 8-2 that CO_2 finial mass distribution would exist in the SV1.5, the filling tube of the filling system and the TTCS loop just like shown in Figure 8-3 with ignoring leak mass during filling process.

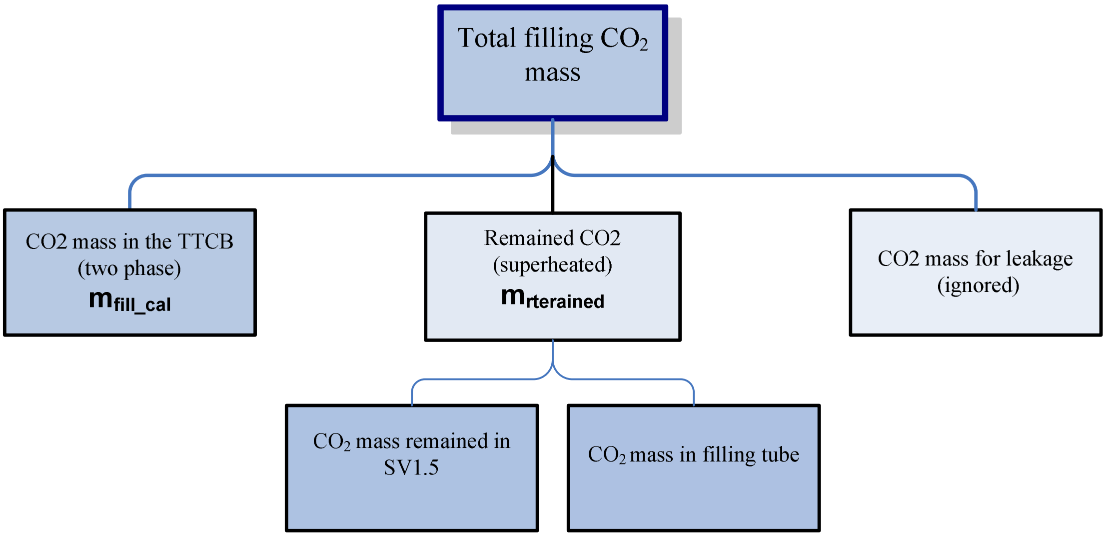

Figure 8-3 CO_2 mass distribution between the filling system and the TTCS loop after finishing filling

8.5 Calculation example

1) Calculation of M_{C_L}

Assumed : V_{Loop}= 1.55L

So

M_{C_L} = FR×V_{Loop}

= 569.6g/L×1.55L

= 882.88g

The above calculated CO_2 must be filled into the TTCS regardless of what temperature you choose for the filling.

2) Calculation of M_{C_R}

If we choose 25°C for the filling, so the retained mass of CO_2 in the filling system including filling box, two extended tube and the stand vessel can be calculated as below:

Known conditions: **T_{Loop}(chosen)**= 22°C, **P(corresponds toT_{accu}(chosen))**=5.995MPa,

T_{VS}(controlled)=30^{o}C,

Once **T_{Loop}(chosen)** is fixed, the final pressure in the filling system and the TTCS loop is also fixed. If the chosen T of CO_2 is 22^{o}C, the corresponding saturated pressure is 5.995MPa.

And in the filling box, the SV1.5 and twos extend tubes

ρ_G (30^{o}C, 5.995MPa) = 170.8g/ L

Solution:

And if volume of the used SV1.5 **V_{VS}** is 1.485L , and filling tube of the filling system **V_{FT}** is 0.042Liter, so the CO2 retained in the SV1.5, the filling box and two filling tubes can be calculated as:

$M_{C\_R} = M_{C\_R\_VS} + M_{C\_R\_FT}$

$= V_{vs} \times \rho_G$ (30^{o}C, 5.995MPa) $+ V_{FE} \times \rho_G$ (30^{o}C, 5.995MPa)

= (1.485L+0.042L)* 170.8g/ L

= 260.82g

According to the above calculation, the temperature chosen for filling will influence the retained mass of CO2 M_{C_R} in the filling system, not influence the mass of CO_2 to be filled into TTCB.

3) Calculation of the total mass

By ignoring the leak mass, the CO_2 mass must filled into the used SV1.5 is

M_{C_L}+ M_{C_R}=882.88g+260.82g =1143.7g

And it pluses net mass of the used SV1.5 **M_{E_VS}** is

$M_{C\_VS} = M_{E\_VS} + M_{C\_L} + M_{C\_R}$

=2747.23g+882.88g+260.82g =3890.83g

Finally, shown by electronic balance, the total mass should be 3890.83g.

Table 10 saturated properties of CO_2 from 15°C to 29°C with interval of 1°C

Temp [^{o}C]	Pressure[MPa]	Density (L) [kg/m^3]	Density (V) [kg/m^3]	Enthalpy (L) [kJ/kg]	Enthalpy (V) [kJ/kg]	Entropy (L) [kJ/K-kg]	Entropy (V) [kJ/K-kg]
15.00	5.081	821.8	159.6	240.1	418.0	1.136	1.754
16.00	5.204	813.0	165.4	243.1	416.6	1.146	1.746
17.00	5.330	803.9	171.5	246.2	415.0	1.156	1.738
18.00	5.458	794.4	178.0	249.4	413.4	1.167	1.730
19.00	5.589	784.5	184.9	252.6	411.6	1.177	1.721
20.00	5.722	774.2	192.3	256.0	409.7	1.188	1.712
21.00	5.857	763.3	200.2	259.5	407.6	1.199	1.703
22.00	5.995	751.8	208.8	263.0	405.3	1.211	1.693
23.00	6.136	739.5	218.1	266.8	402.8	1.223	1.682
24.00	6.279	726.3	228.3	270.7	400.0	1.236	1.670
25.00	6.425	712.0	239.6	274.9	396.8	1.249	1.658
26.00	6.574	696.2	252.4	279.3	393.2	1.263	1.644

27.00	6.727	678.4	267.3	284.2	389.0	1.279	1.628
28.00	6.882	657.5	285.1	289.7	384.0	1.296	1.609
29.00	7.042	631.7	307.8	296.3	377.5	1.317	1.586

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